

# Biosorption of Congo Red from aqueous solution onto burned root of *Eichhornia crassipes* biomass

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**Abstract** Biosorption is becoming a promising alternative to replace or supplement the present dye removal processes from dye containing waste water. In this work, adsorption of Congo Red (CR) from aqueous solution on burned root of *Eichhornia crassipes* (BREC) biomass was investigated. A series of batch experiments were performed utilizing BREC biomass to remove CR dye from aqueous systems. Under optimized batch conditions, the BREC could remove up to 94.35 % of CR from waste water. The effects of operating parameters such as initial concentration, pH, adsorbent dose and contact time on the adsorption of CR were analyzed using response surface methodology. The proposed quadratic model for central composite design fitted very well to the experimental data. Response surface plots were used to determine the interaction effects of main factors and optimum conditions of the process. The optimum adsorption conditions were found to be initial CR concentration = 5 mg/L<sup>-1</sup>, pH = 7, adsorbent dose = 0.125 g and contact time = 45 min. The experimental isotherms data were analyzed using Langmuir, Freundlich, Temkin and Dubinin–Radushkevich (D–R) isotherm equations and the results indicated that the Freundlich isotherm showed a better fit for CR adsorption. Thermodynamic parameters were calculated from Van't Hoff plot, confirming that the adsorption process was spontaneous and exothermic. The high CR adsorptive removal ability and regeneration efficiency of this adsorbent suggest its applicability in industrial/household systems and data

generated would help in further upscaling of the adsorption process.

**Keywords** Adsorption · Congo Red · *E. crassipes* biomass · Central composite design · Regeneration

## Introduction

The release of huge volume of dyes containing waste water from different industries like textile, leather, paper, rubber, printing, food and cosmetics is a dangerous global concern. These harmful dyes containing waste water are disposed directly into the river without proper treatment causing carcinogenic and mutagenic effects (Akar and Özcan 2009). Therefore, the massive development towards remediation of such problems is highly desirable (Ahmad and Alrozi 2011). Nowadays, the effect of harmful dyes is a serious health problem due to their negative impact on both flora and fauna (Saha et al. 2010). Akar and Özcan (2009) reported that the breakdown products of dyes are dangerous and cause serious health problems to both humans and animals.

Congo Red is an anionic azo dye which is highly toxic to the living body. This dye has been known to cause an allergic reaction. Its decomposition results in carcinogenic products. It acts as a skin, eye and gastrointestinal irritant. It affects blood factors such as clotting and induces drowsiness and respiratory problems (Demirbas 2009). Congo Red has a strong affinity to cellulose fibers. It has been recognized as a serious community health problem because of its toxic nature and therefore, its removal is highly essential.

Development of effective technologies to remove dyes from water drew great attention over the last two decades. At present, several physicochemical and biological methods are used for decolorization of waste water such as ion

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exchange, lime softening, membrane filtration, coagulation, precipitation, advanced oxidation process (Roy and Mondal 2014) and adsorption (Diouri et al. 2015). Among these, most of the techniques have been restricted due to high consumption or expensive chemicals and after removal generate new type of toxic sludge. Therefore, there is an urgent requirement for development of innovative method. A simple and cost-effective adsorbent was used by Roy et al. (2013) in their earlier work. Previous literature demonstrated many adsorbents for removal of organic dye. However, the most important dye adsorbent is granular activated carbon. Activated carbon adsorption method is a method applied earlier, and this method is very effective for the removal of dissolved organic matter. Many other materials like tree fern (Ho et al. 2005), orange peel (Arami et al. 2005), banana pith (Namasivayam et al. 1997), date pits (Fawzi et al. 2003), peanut hulls (Gong et al. 2005), giant duck weed (Waranusantigul et al. 2003), teak tree bark powder (Patil et al. 2011), corn cobs and barley husk (Chandran et al. 2002), rice husk and saw dust (Vadivelan and Kumar 2005; Malik 2003), *Agave americana* (L.) fibers (Ben Hamissa et al. 2007) have been tested for their dye adsorption capacity. The search for an appropriate and inexpensive biomass is a continuing process. The most effective and optimized utilization of a biomass demands a detailed understanding of the binding mechanism.

*Eichhornia crassipes* is an aquatic plant which is widely available in India, is listed as one of the most productive plants on earth and is considered one of the world's worst aquatic plants. It forms dense mats that interfere with navigation, recreation, irrigation, and power generation. These mats competitively exclude native submersed and floating-leaved plants. Due to the growth rate of *E. crassipes*, which is extremely high (Martina et al. 2004; Mall et al. 2005), burned root of *E. crassipes* (BREC) has attracted the special attention of scientists to use it as a potential biosorbent. So, this biomass can also be utilized to remove CR from aqueous solutions which formed the motivation of this study. Only a few studies on *E. crassipes*, as an adsorbent, have been conducted (Wanyonyi et al. 2014; Soni et al. 2012) and no research has been reported on the burned root of *E. crassipes* (BREC) that may have higher CR removal efficiency.

Thus, the purpose of this study is to investigate the application of BREC for the removal of CR from aqueous system. The solute and solvent interaction mechanism were explained by adsorption isotherm. Various kinetic models were used to determine the rate and order of the adsorption process. A central composite design (CCD) in response surface methodology (RSM) by Design Expert Version 7.0.3 [Stat-Ease, Inc. (2009), USA] is used to optimize the process parameters for the biosorption process. The applied

models have also been investigated to study the individuals as well as the combined effect of different variables influencing the adsorption process. When compared with other similar adsorption studies, the novelty of using RSM in this work is in reducing the number of experiments, studying the effect of factor interactions and developing a statistical mathematical model equation.

## Materials and methods

### Reagents and apparatus

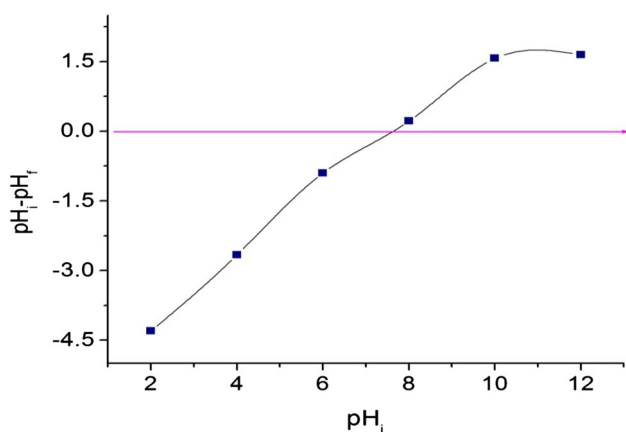
All chemicals were used of analytical grade. Congo Red (CR), the typical anionic dye was selected as the adsorbate in the present study. All reagents and standards were prepared using double distilled water. The required CR concentrations to perform the whole experiments were prepared by the dilution of main solution in required ratio. The pH of CR solution was adjusted by the addition of 0.1 M HCl and NaOH solutions whenever necessary. Experimentally, the zero point charge of root of *E. crassipes* was determined by a series of pH from 2 to 12 by 0.1 (M) KNO<sub>3</sub> and KOH solution. All glassware were cleaned by being soaked in 15 % HNO<sub>3</sub> and rinsed with double distilled water before use. The batch experiments were conducted in a temperature controlled magnetic stirrer (Spinot, Cat No. 6030). A UV–visible spectrophotometer (Systronics, Vis double beam Spectro 1203) with a 1-cm quartz cell was used for quantitative determination of CR in solution. pH was measured by a digital pH meter (Eutech, pH 700 Meter) with an accuracy of 0.01 unit. To optimize the process parameters for the adsorption process, a central composite design (CCD) in response surface methodology (RSM) by Design Expert Version 7.0.3 [Stat-Ease, Inc (2009), USA] is used.

### Adsorbent preparation

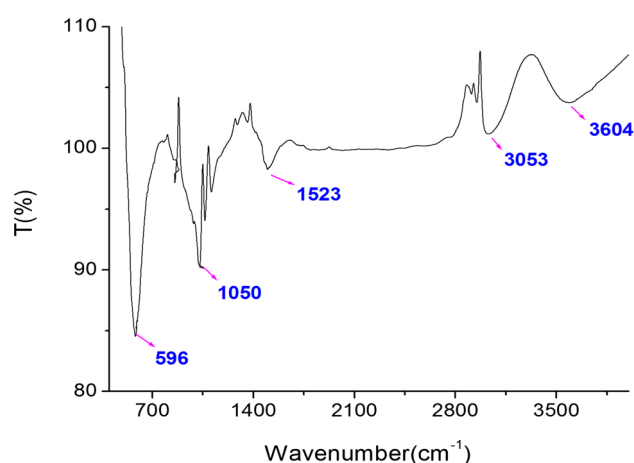
*Eichhornia crassipes* (*E. crassipes*) was collected from a local pond of Golapbag campus of Burdwan University, Burdwan, West Bengal, India. The roots of *E. crassipes* were separated and extensively washed with tap water to eliminate soil dust and earthy materials. Finally roots were washed several times with distilled water and sliced in pieces manually and dried at 50 °C in an oven. The dried roots were burned in muffle chamber at 500 °C and stored in airtight container for further use as biosorbent.

### Characterization of prepared biosorbent

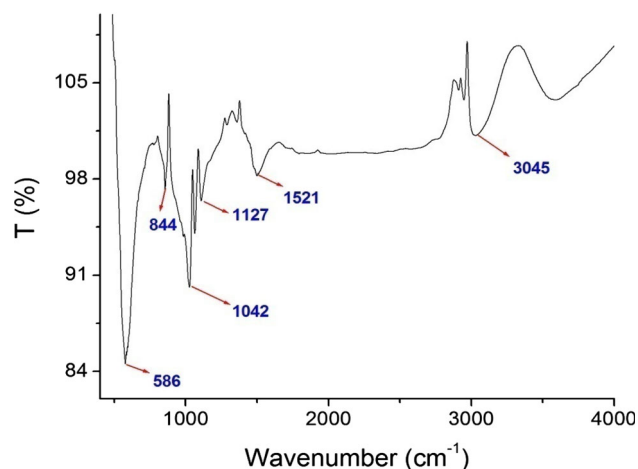
The stable form of BREC in water, dilute acids and bases, behaves as neutral at pH zero charge. The point of zero



**Fig. 1** pH of zero point charge of BREC

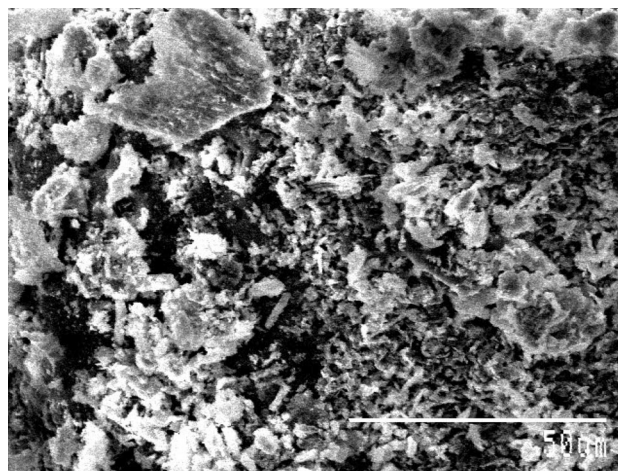


**Fig. 2** FTIR spectrum of BREC (before treatment)

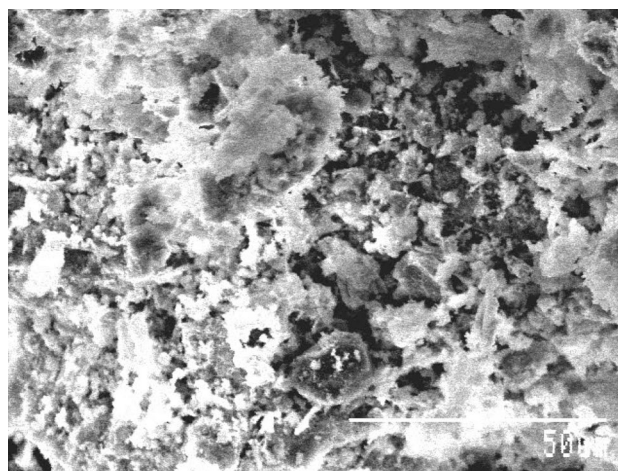


**Fig. 3** FTIR spectrum of BREC (after treatment)

charge is 7.59 (Fig. 1) irrespective of difference in concentration of  $\text{HNO}_3$  used. Characterization of the prepared BREC was done by FTIR and SEM analysis is shown



**Fig. 4** SEM micrograph of BREC (before treatment)



**Fig. 5** SEM micrograph of BREC (after treatment)

consequently in Figs. 2, 3, 4 and 5, respectively. FTIR spectroscopy is a very useful tool for investigating the band positions and absorption peaks. FTIR analysis was performed using Perkin-Elmer FTIR (FTIR spectrum RX1). The FTIR spectra of BREC before and after treatment at  $250^\circ\text{C}$  are shown in Figs. 2 and 3, respectively. The absorption band observed at  $586\text{ cm}^{-1}$  is attributed to the C–Br stretching vibrations. The band at  $1042\text{ cm}^{-1}$  may be attributed to C–N stretching frequencies and the band at  $1127\text{ cm}^{-1}$  may be attributed to C–O– species in the film. The band at  $1521\text{ cm}^{-1}$  may be assigned to N–H and the band at  $3045\text{ cm}^{-1}$  may be attributed to alkenes C–H stretching frequency.

Scanning electron microscopy (SEM) analysis is a useful tool for the study of the surface morphology of an adsorbent. The SEM micrographs (25 kV; 50 mm) at 1000 magnification for BREC surface before and after CR adsorption at the optimization conditions are shown in

Figs. 4 and 5, respectively. Figure 4 clearly shows the presence of porous, rough, and irregular surface morphology of the unloaded BREC. The surface roughness of an unloaded adsorbent indicated the availability of a tremendous surface area of BREC meant for high adsorption capacity in the adsorption process (Roy et al. 2013). Contrary to this, after adsorption, the adsorbent surface seemed to have decreased porosity due to the heavy impregnation of CR onto the surface of the BREC (Fig. 5).

### Batch adsorption procedure

In the batch adsorption experiments, the effect of different parameters (i.e., pH, contact time, initial CR concentration, dose of biosorbent, and temperature) on adsorption of CR was studied. The 100 ml solution of CR was taken in each Borosil beaker of volume 250 ml separately. After pH adjustment, a known quantity of biosorbent (BREC) was added and the CR bearing suspensions was kept under magnetic stirring until the equilibrium was reached. At predetermined times, 5 ml of reaction mixture was collected and centrifuged (4000 rpm, 15 min) in a centrifuge. The absorbance at 497 nm wavelengths of the supernatant was determined using ultraviolet–visible spectrophotometer. Dye concentration in the reaction mixture was calculated from the calibration curve. Adsorption experiments' curves were conducted by varying initial concentration of the dye, pH, contact time, and biosorbent dose under the aspect of adsorption kinetics. The amount of dye adsorption by BREC powder at time  $t$ ,  $q_t$  (mg/g) was calculated by the following mass balance relationship:

$$q_t = \frac{(c_0 - c_t) \times V}{m} \quad (1)$$

The dye removal efficiency i.e., % of adsorption was calculated as:

$$\% \text{ of adsorption} = \frac{(c_0 - c_t) \times 100}{c_0} \quad (2)$$

where  $C_0$  is the initial dye concentration (mg/L),  $C_t$  is the concentration of the dye at any time  $t$ ,  $V$  is the volume of solution (L) and  $m$  is the mass (g) of biosorbent.

### Desorption experiments

Desorption experiments were conducted by either HCl or NaOH as a desorbing agent. Primarily, 125 mg of fresh adsorbent was added to 100 ml of 5 mg/L CR solution at pH 7.0 and shaken for 12 h. Next, the residual CR concentration was determined using spectrophotometer as described earlier and the CR-adsorbed BREC was separated by centrifugation. The CR-loaded adsorbent was washed gently with water to remove any unadsorbed dye

and dried. The desorption process was performed by mixing the dried adsorbent with 50 ml of distilled water at different pH values (4.0–10.0). After the mixture was shaken for a predetermined time, the desorbed CR concentration was determined spectrophotometrically. The desorption efficiency of CR was calculated as the ratio of the desorbed amount ( $q_{de}$ ) to the adsorbed amount ( $q_{ad}$ ).

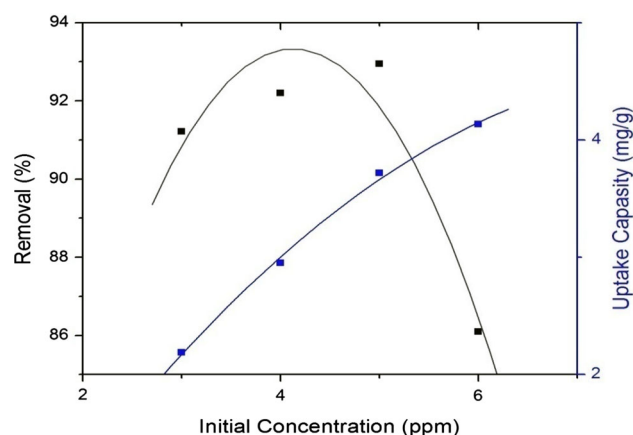
$$\text{Desorption efficiency } (\%) = \frac{q_{de}}{q_{ad}} \times 100$$

## Results and discussion

### Batch adsorption study

#### Effect of initial concentration

The adsorption behavior of CR was studied in the concentration range of 3–6 mg/L initially at pH 7.0. The amount of dye adsorbed by BREC increases from 2.25 to 4.45 mg/g<sup>−1</sup> when increasing the initial dye concentration from 3 to 6 mg/L<sup>−1</sup> (Fig. 6). The performance of dye removal efficiency of BREC increases with increasing dye concentration is probably due to increase of driving force to overcome all mass transfer resistances of the dye molecules between the aqueous and solid phases, leading to an increasing equilibrium sorption until saturation is achieved (Wanyonyi et al. 2014). The percentage was greater at lower initial concentration and smaller at higher initial concentration. At lower concentration, the ratio of amount of CR available and the number of adsorption dyes was low, hence comparatively, a higher percentage of adsorption resulted. At higher concentrations, this ratio progressively increased due to number of adsorption sites becoming fewer compared to the amount of CR available.



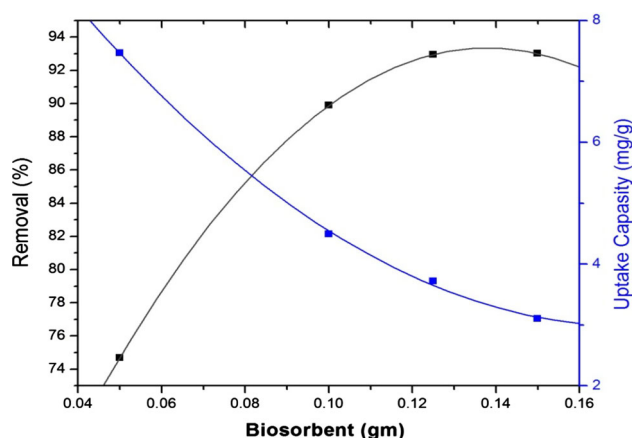
**Fig. 6** Effect of initial concentration on the adsorption of Congo Red by BREC: contact time 20 min; dose 0.05 g; pH 6.5; temperature 25 °C; agitation speed 300 rpm



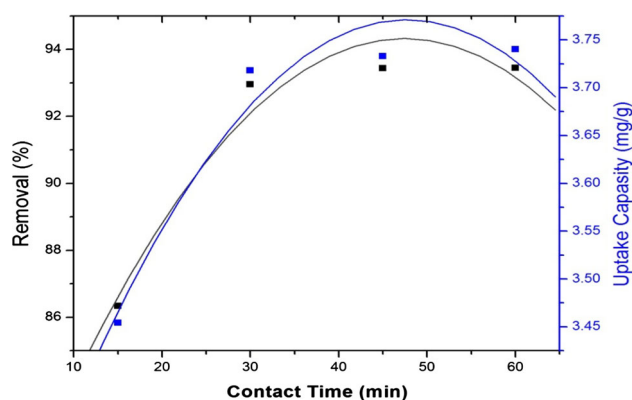
Therefore, a decreasing trend in percentage adsorption from a lower concentration to a higher concentration was recorded (Okeola and Odebunmi 2010; Chen et al. 2003) (Figs. 7, 8).

### Effect of pH

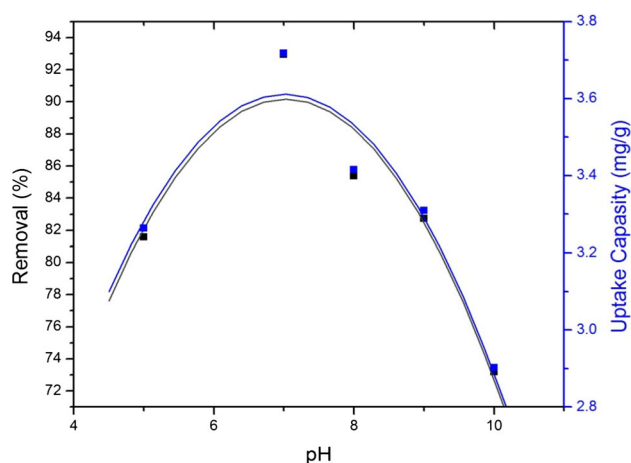
Figure 9 represents the percentage removal as a function of the pH at optimum concentration (5 mg/L) of CR. The maximum adsorption of 94.35 % was obtained at the pH of 7.0 and all further experiments were carried out at pH 7.0. The efficiency of removal processes strongly depends upon the pH of the reaction solution. The adsorbent behaves as neutral at pH zero charge. Adsorption of cation is favored at  $\text{pH} > \text{pH}_{\text{zpc}}$ , while the adsorption of anion is favored at  $\text{pH} < \text{pH}_{\text{zpc}}$  (Mondal 2009). The point of zero charge is 7.59 (Fig. 1). So, adsorption capacity of anionic dyes (mainly sulfonated dyes) such as Congo Red reaches a maximum value below the zero point charge at  $\text{pH} < \text{zpc}$ .



**Fig. 7** Effect of biosorbent dose on the adsorption of Congo Red by BREC: contact time 20 min; initial concentration 5 mg/L; pH 6.5; temperature 25 °C; agitation speed 300 rpm



**Fig. 8** Effect of contact time on the adsorption of Congo Red by BREC: dose 0.125 g; initial concentration 5 mg/L; pH 6.5; temperature 25 °C; agitation speed 300 rpm



**Fig. 9** Effect of pH on the adsorption of Congo Red by BREC: dose 0.125 g; contact time 45 min; initial concentration 5 mg/L; temperature 25 °C; agitation speed 300 rpm

Figure 9 indicated that the CR adsorption rates increased with increasing pH up to 7 and further increase in pH, no significant change in dye removal efficiency was observed. Therefore, the best pH of the treated solution should be limited to this value, when using *BREC*, because the electrostatic interaction between the anionic dye and positive surface of adsorbent is enhanced (Daneshvar et al. 2007; Khodja et al. 2001). In very low pH range, the anionic dyes were converted into neutral species by the protonation of dyes. Therefore, neutral dyes are less attracted with cationic biosorbent. At pH above the  $\text{pH}_{\text{zpc}}$ , the number of negatively charged sites on the biosorbent surface increased. A negatively charged surface site is not favored due to the increased electrostatic repulsion. However, it was observed that at neutral pH range 7, significant dye adsorption on *BREC* takes place. Almost similar results were reported for biosorption of CR from aqueous solution onto different biosorbents (Ghribi et al. 2014; Wanyonyi et al. 2014; Hu et al. 2010; Mall et al. 2005).

### Effect of adsorbents' dose

The effect of adsorbent dose on CR adsorption is shown in Fig. 7. It determines the adsorption capacity of CR. The removal of CR was studied at different doses (0.5–1.5 gm/L) by keeping the contact time 45 min, pH 7, concentration 5 ppm. It is apparent from Fig. 7 that the removal process was increased from 74.69 to 94.35 % when *BREC* dose was increased from 0.05 to 0.125 for 100 ml solution. It is perhaps due to that the number of available active sites and surface area increased by increasing the adsorbent dose. Above the dose of 0.125 gm/100 ml, there was no remarkable change in percentage of removal of CR dye. It might be due to overlapping of active sites on biosorbents. At the same time, the adsorbed CR amount at equilibrium

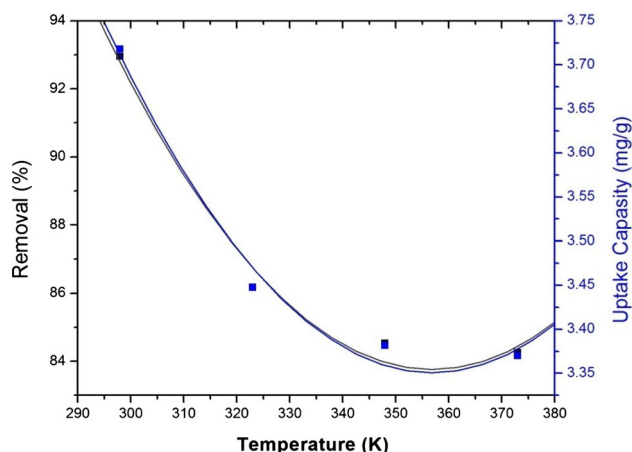
( $q_e$ , mg/g) decreased with increasing adsorbent dosage because of the split in the flux or the solute concentration gradient between the solution and the adsorbent surface (Kumar et al. 2010; Vadivelan and Kumar 2005).

#### Effect of contact time

The CR removal process was a function of contact time. The uptake capacity of CR dye by *BREC* dose was very fast and 86 % removal was sorbed within 15 min and equilibrium was established after 45 min. From Fig. 8 it is clear that the sorption was fast initially and became slow at later stages till the equilibrium was attained. The rapid adsorption at the initial phase is probably due to the availability of active sites on the catalyst surface (Das et al. 2013; Saqib et al. 2013). However, with a lapse of time, the remaining unsaturated sites cannot be occupied because of repulsive force between the solute (CR) on the solid and bulk phases (Roy et al. 2013; Sokker et al. 2011). Similar results have been previously reported in the literature for other dye removal also (Chanzu et al. 2012).

#### Effect of temperature

The effect of temperature on the removal of CR by *BREC* was determined at different temperatures from 25 to 90 °C (Fig. 10). When the temperature was increased from 25 to 90 °C the percent removal of CR was decreased from 94.35 to 84.24. These results show that the adsorption of CR onto *BREC* is an exothermic process and the physical bonding between the dye molecules and the active sites of the *E. crassipes* weaken with increasing temperature. Similar results were reported by Hu et al. (2010) who investigated the effect of temperature on removal of Congo Red from aqueous solution by cattail root and indicated that



**Fig. 10** Effect of temperature on the adsorption of Congo Red by *BREC*: dose 0.125 g; initial concentration 5 mg/L; contact time 45 min; pH 7.0; agitation speed 300 rpm

adsorption decreases with increasing temperature. However, opposite trend of dye adsorption with temperature is also explained that at high temperature, solutes move with greater speed, therefore, less time required for interaction with the active sites of the adsorbent (Kanwal et al. 2012; Samani et al. 2010).

#### Interaction of variables

In order to study the interaction among the different independent range of the variables in Table 5 and their corresponding effect on the response, contour plots were drawn (Figs. 18, 19, 20, 21). Contour plot is the projection of the response surface as a two-dimensional plane. This analysis gives a better understanding of the influence of variables, and their interaction on the response as a function of two variables at a time. The interaction between dye molecules and the surface of the biosorbent absolutely depends on their charges (Maurya et al. 2006). The entire results are shown in contour plots (Fig. 19). On the other hand, Fig. 21 depicts the interaction effect of contact time and pH on the removal process. Within the experimental range, the response function, i.e., the removal increases with increasing contact time. The percentage removal increases with increase in both the initial concentration, pH as well as biosorbent dose up to the optimum value and decreases thereafter. Such behavior can be explained in the sense that all biosorbents have a limited number of active sites which become saturated at a certain concentration (Das et al. 2013; Chowdhury et al. 2011).

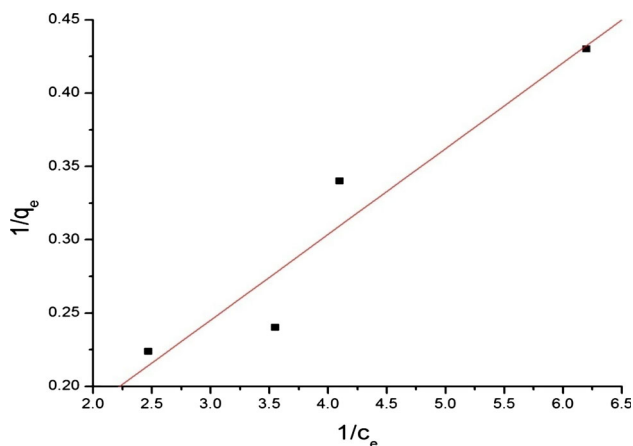
The influence of the initial pH of the CR solution on its adsorption on *BREC* is shown in Fig. 20. The removal efficiency increased from 82.97 to 94.35 with increase in pH of the dye solution, appreciably up to pH 7.0. With further increase in pH, no significant change in dye removal efficiency was observed. The maximum adsorption was observed at pH 7.0. This pH effect can be explained by the different interaction between CR and roots of *E. crassipes* in terms of surface charge, degree of ionization and speciation. Moreover, higher adsorption of CR dye is due to strong electrostatics attraction between anionic CR dye molecules and cationic biosorbent. Identically, the  $q_e$  values were found to decrease in the alkaline pH range of 7.0–10. Similar results were reported by earlier researchers (Vijaraghavan et al. 2004).

In addition, CR shows a fast rate of adsorption during the first 15 min of the dye–sorbent contact. The fast adsorption rate is due to the large amount of surface area available for adsorption of the dye molecules. At higher contact time the rate of adsorption decreases, gradually leading to equilibrium (Chakraborty et al. 2011) and less available binding sites (Saha et al. 2010).

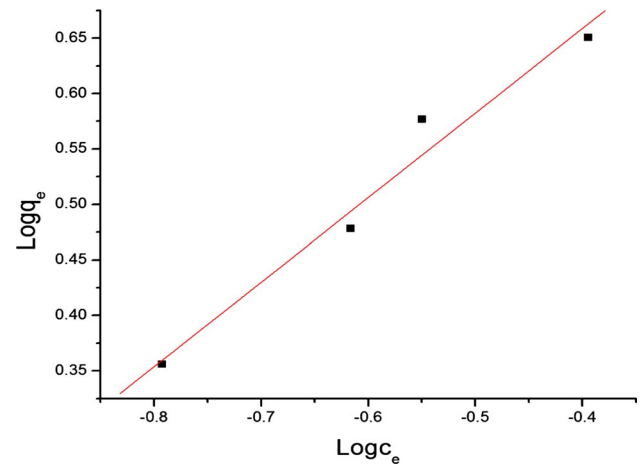
A literature review shows that only a few works on burned root of *E. crassipes* have been reported, otherwise most of the studies for CR removal have been conducted by simple biosorbent. The higher equilibrium uptake capacity of CR makes the adsorbent attractive for CR removal filter units. Therefore, the CR uptake capacity on *BREC* is compared with previously reported other adsorbents (Table 4). The results show the applicability of *BREC* for the removal of CR from aqueous solutions using a significance study.

### Equilibrium isotherms

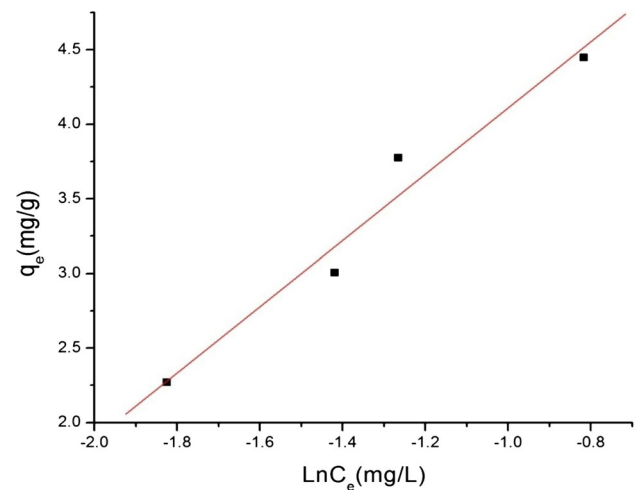
An adsorption isotherm represents the equilibrium relationship between the adsorbate concentration in the liquid phase and that on the adsorbents surface at a given condition. A number of isotherms have been developed to describe equilibrium relationships. In the present study, the Langmuir, Freundlich, Temkin and Dubinin–Radushkevich (D–R) models were used to describe the equilibrium isotherms. The illustration of the equilibrium adsorption plots and the summarized isotherm models are shown in Figs. 11, 12, 13, 14 and Table 1. From Table 1, it is observed that the Freundlich isotherm showed good fit to the experimental equilibrium adsorption data than the Langmuir, Dubinin–Radushkevich and Temkin isotherm equations for CR adsorption according to the values of  $R^2$ . It is also seen from Table 1 that the Langmuir maximum adsorption capacity  $q_{\max}$  (mg/g) is 14.49 and the equilibrium constant  $K_L$  (L/mg) is 1.189. The separation factor ( $R_L$ ) values are 0.218, 0.173, 0.143 and 0.122 while initial CR concentrations are 3, 4, 5 and 6 mg/L, respectively. All the  $R_L$  values were found to be less than one and greater than zero indicating the favorable adsorption of CR by *BREC*. The Freundlich constant  $K_F$  and  $n$  are constants



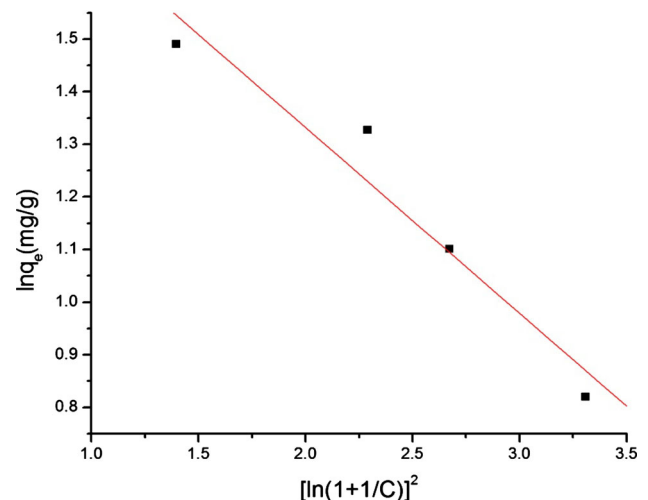
**Fig. 11** Langmuir isotherm for adsorption of Congo Red onto *E. crassipes* biomass



**Fig. 12** Freundlich isotherm for adsorption of Congo Red onto *E. crassipes* biomass



**Fig. 13** Temkin isotherm for adsorption of Congo Red onto *E. crassipes* biomass



**Fig. 14** D–R isotherm for adsorption of Congo Red onto *E. crassipes* biomass

**Table 1** Isotherm data for adsorption of CR dye by BREC

Adsorption isotherm	Equations	Parameters (unit)	Values	$R^2$
Langmuir isotherm	$\frac{1}{q_e} = \frac{1}{q_{\max} K_L C_e} + \frac{1}{q_{\max}}$	$q_{\max}$ (mg/g)	14.49	0.912
		$K_L$ (L/mg)	1.189	
Freundlich isotherm	$\log q_e = \log k_F + \frac{1}{n} \log C_e$	$K_F$ (mg/g)	9.18	0.970
		$(L/mg)^{1/n}$	1.3	
		n		
Temkin isotherm	$q_e = \frac{RT}{b_T} (\ln A_T + \ln C_e)$	$A_T$ (L/mg)	707.9	0.963
		$b_T$ (KJ/mol)	1.116	
D-R isotherm	$\ln q_e = \ln q_{\max} - \frac{1}{2E^2} \times \left[ RT \ln \left( 1 + \frac{1}{C_e} \right)^2 \right]$	$q_{\max}$ (mg/g)	7.68	0.941
		$E$ (KJ/mol)	2.948	

$q_{\max}$  is the maximum adsorption capacity;  $K_L$ ,  $K_F$ ,  $A_T$  and  $b_T$  are different adsorption constants; n is the heterogeneity factor; E is the mean free energy of adsorption per mole of the adsorbate; T is the temperature (K), and R is the ideal gas constant (8.3145 J/mol K)

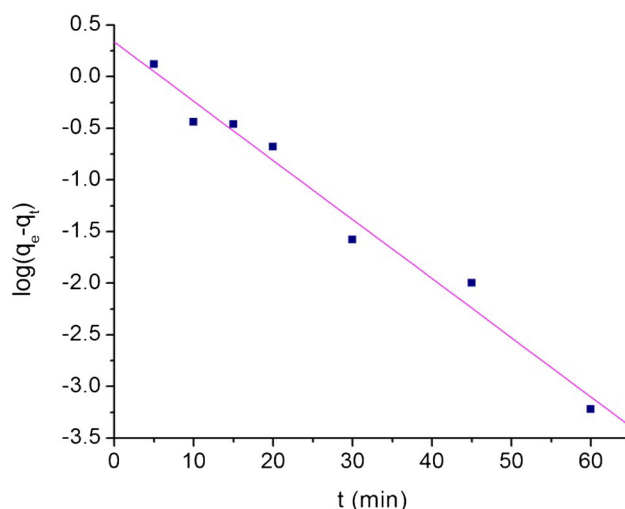
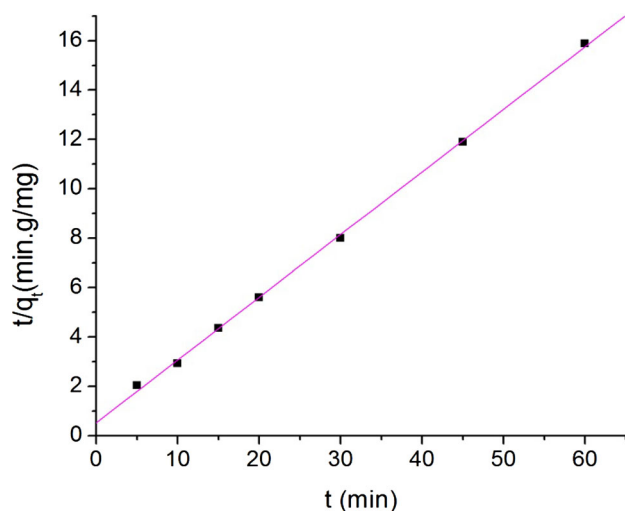
incorporating the factors affecting the adsorption capacity and intensity of adsorption, respectively. The adsorption capacity of the adsorbent and the value of  $K_F$  is 9.18 (mg/g)  $(L/mg^{-1})^{1/n}$ . Furthermore, the value of 'n' at equilibrium is 1.30. The value of n lies in between 1 and 10 also represents a favorable adsorption. From Temkin constant,  $b_T$  related to adsorption binding energy for CR is found 1.116 kJ/mol. In addition, D–R isotherm shows the value of the adsorption energy (E) is very high 2.948 kJ/mol. So it can thus be concluded that this biosorption process will play a dominating role to remove the CR dye from aqueous solution onto BREC (Dada et al. 2012; Samarghandi et al. 2009).

### Adsorption kinetics

The pseudo-first-order and pseudo-second-order kinetic models were tested to investigate the rate of biosorption of Congo Red on BREC biomass (Figs. 15, 16). The linearized form of biosorption kinetics (Guo et al. 2002) and their constants are presented in Table 2. The plot of  $t/q$  versus t shows a linear relationship which confirmed that the biosorption of CR on BREC biomass followed the pseudo-second-order reaction and is shown in Fig. 16. The values of  $q_s$  and  $k_2$  can be determined from the slope and intercept of the plot. It is clear that the pseudo-second-order kinetic model showed excellent linearity with high correlation coefficient ( $R^2 = 0.999$ ) at all the studied concentrations in comparison to the first-order kinetic model (Table 2). In addition, the calculated  $q_s$  values also agree with the experimental data in the case of pseudo-second-order kinetic model.

### Thermodynamic parameters

Thermodynamic parameters can be determined from the thermodynamic equilibrium constant  $K_0$ , where  $K_0$  is the

**Fig. 15** First-order kinetic model for adsorption of Congo Red onto *E. crassipes* biomass**Fig. 16** Second-order kinetic model for adsorption of Congo Red onto *E. crassipes* biomass



**Table 2** Summary of parameters for various kinetic models

Kinetic model	Equations	Parameters (unit)	Values	$R^2$
Pseudo first-order	$\ln(q_e - q_t) = \ln q_e - K_1 t$	$q_e$ (μg/g) $K_1$ (min <sup>-1</sup> )	2.15 0.1313	0.977
Pseudo second-order	$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e}$	$q_e$ (μg/g) $K_2$ (g/μg · min)	3.95 0.1196	0.999

$q_t$  is the adsorption capacity at time  $t$ ;  $k_1$  and  $k_2$  are the first-order and second-order rate constants, respectively

**Table 3** Thermodynamic parameters for adsorption of CR dye by BREC

Thermodynamic parameters	Equations	Values (J/mol)
Standard free energy	$\Delta G^0 = -RT \ln K_c$	
298 K		-6421
323 K		-4645
348 K		-4590
373 K		-4843
Standard enthalpy change	$\ln K_c = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT}$	-12.346
Standard entropy change		-21.525

$K_c$  is the equilibrium constant;  $T$  and  $R$  are Kelvin temperature and molar gas constant, respectively

ratio of adsorption capacity (mg/g<sup>-1</sup>) and concentration at equilibrium state. The thermodynamic parameters ( $\Delta G$ ,  $\Delta H$  and  $\Delta S$ ) of the adsorption of CR on BREC were calculated using equations and  $\Delta G = -RT \ln K_0$ , where  $R$  is the ideal gas constant,  $T$  is the temperature (K) and  $K_0$  is the distribution coefficient calculated from the experiment. The values of  $\Delta H$  and  $\Delta S$  were calculated from the slope and intercept of the Van't hof plots listed in Table 3. The negative values of free energy,  $\Delta G$  indicate the feasibility

of the process and its spontaneity in nature (Table 3). Moreover, the negative values of  $\Delta H$  also suggest that the adsorption process is exothermic in nature. On the other hand, the negative value of  $\Delta S$  indicates that the randomness decreases with progress of the adsorption process. Therefore, the thermodynamic parameters for the CR adsorption showed good agreement with previous literature (Vijaykumar et al. 2012; Achmad et al. 2012) (Tables 4, 5).

### Central composite design analysis

CCD is a statistical method based on the multivariate nonlinear model that has been widely used for the optimization of process variables of biosorption and also to determine the regression model equations and operating conditions from the appropriate experiments (Kalavathy et al. 2009; Arulkumar et al. 2011). Generally, a system or process with several variables is likely to be driven primarily by some principal factors and low-order interactions. In the present work, only two-way interactions were investigated. Linear, two-factor interaction (2FI), quadratic and cubic models were used to analyze the experimental data to obtain the appropriate regression equations. To

**Table 4** Comparison of adsorption capacities of various adsorbents for Congo Red

Adsorbent	Adsorption capacity (mg/g)	Temperature (0 °C)	pH	References
Kaolin	5.60	25–60	3–11	Zhang et al. (2007)
Acid-activated red mud	7.08	–	7	Salleh et al. (2011)
Australian kaolins				
Q38	5.62	25–60		Abd El-Latif et al. (2010)
K15GR	6.92			
Ceram	8.97			
Sandy loam, loam and clay Greek soils	0.500–0.625	–	4–12	Ghaedi et al. (2012)
Cambic chernozem soil (Romanian)	2.24	–	–	Purkait et al. (2007)
Kaolin	5.44	25–60	7.5 ± 0.3	Vimonses et al. (2009)
Waste red mud	4.05	–	2	Lorenc-Grabowska et al. (2007)
Marine algae ( <i>Valoria bryopsis</i> )	3.40–10.5		1–7	
Activated carbon from coir pith	6.72	35	7.58–8.19	Namasivayam and Kavitha (2002)
Zeolite	4.30	25–60	3–11	Zhang et al. (2007)
Burned root of <i>E. crassipes</i> (BRE C)	4.88	25	7.0	This study

**Table 5** Variables and levels considered for the adsorption of Congo Red onto burned root of *E. crassipes* biomass

Name (factor)	Units	Low	High
Initial concentration ( $X_1$ )	ppm	3	6
pH ( $X_2$ )		5	10
Adsorbent dose ( $X_3$ )	gm	0.05	0.125
Contact time ( $X_4$ )	minute	15	60

determine the adequacy of the models depicting the removal of CR by *BREC* biomass, two different tests, i.e., the sequential model sum of squares and the model summary statistics were conducted. The corresponding results are tabulated in Tables 6 and 7. The fitness of the model was determined by regression coefficient ( $R^2$ ) and its statistical significance was evaluated by an F test (Peng et al. 2002). The higher the value of  $R^2$ , the better is the model. The results from the sequential model indicated that the 2FI model did not provide a good description of the experimental data.  $R^2$  is a measure of the reduction amount in the variability of the response obtained by the independent factor variables in the model. Furthermore, the quadratic model had maximum “predicted  $R^2$ ” and “adjusted  $R^2$ ” values indicating that the quadratic model provided an excellent explanation for the relationship between the independent variables and the corresponding response. “Adequate precision” measures the signal-to-noise ratio and a ratio  $>4$  is desirable. It is clear that only the “adequate precision” value of the quadratic model indicated an adequate signal, validating the viewpoint mentioned above. Consequently, the quadratic model could be used to

navigate the design space and for this reason it was selected as the most appropriate model for further analysis.

### ANOVA analysis

To detect a valid model, the actual responses were fitted with existing linear, two-factor interactions (2FI), cubic and quadratic model by CCD. The ANOVA results (Table 8) of the quadratic model suggest that the model was highly significant, as it is evident from the Fisher’s  $F$  value (43.11) with a low probability value ( $P < 0.0001$ ). The goodness of fit of the model was further checked by the correlation coefficient ( $R^2$ ) between the experimental and model predicted values of the response variable. The predicted correlation coefficient (pred.  $R^2 = 0.8462$ ) also shows good agreement with the adjusted correlation coefficient (adj.  $R^2 = 0.9531$ ). These models can be evaluated on the basis of scores obtained from the sequential model sum of squares (Table 6), and it showed that the quadratic model had a high score. The larger magnitude of  $F$  (43.11) and smaller value of  $p$  ( $<0.0001$ ) indicated the high significance of the model, and hence the quadratic model was found to be good and selected to continue the progress. The adequacy and significance of the selected model can be further evaluated using the analysis of variance (ANOVA). The evidences were Fisher variation ratio ( $F$  value), probability value ( $p$  value), lack of fit, coefficient of determination  $R$ -squared ( $R^2_d$ ), adjusted  $R$ -squared ( $R^2_{Adj}$ ), predicted  $R$ -squared ( $R^2_{Pred}$ ) and adequate precision. Adequate precision is a signal-to-noise ratio, which compares the range of the predicted values at the design points to the average prediction error. The ratios greater than 4 indicate adequate model discrimination.  $R^2_{Adj}$

**Table 6** Adequacy of the model tested

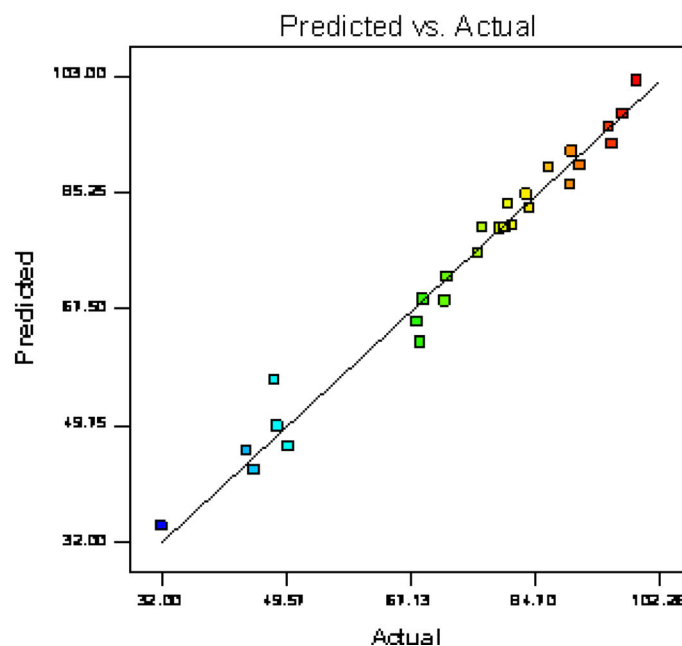
Source	Sum of squares	df	Mean square	$F$ value	$p$ value	Prob $> F$
Mean versus total	1.680E + 005	1	1.680E + 005			
Linear versus mean	7160.09	4	1790.02	24.17	$<0.0001$	
2FI versus linear	364.29	6	60.71	0.78	0.5990	
Quadratic versus 2FI	1269.02	4	317.25	21.77	$<0.0001$	Suggested
Cubic versus quadratic	211.86	10	21.19	15.82	0.0035	Aliased
Residual	6.69	5	1.34			
Total	1.771E + 005	30	5901.72			

**Table 7** Model summary statistics for Congo Red adsorption onto burned root of *E. crassipes* biomass

Source	SD	$R^2$	Adjusted $R^2$	Predicted $R^2$	PRESS	
Linear	8.61	0.7945	0.7616	0.6883	2808.82	
2FI	8.85	0.8349	0.7481	0.7209	2514.96	
Quadratic	3.82	0.9757	0.9531	0.8462	1385.69	Suggested
Cubi	1.16	0.9993	0.9957		Aliased	

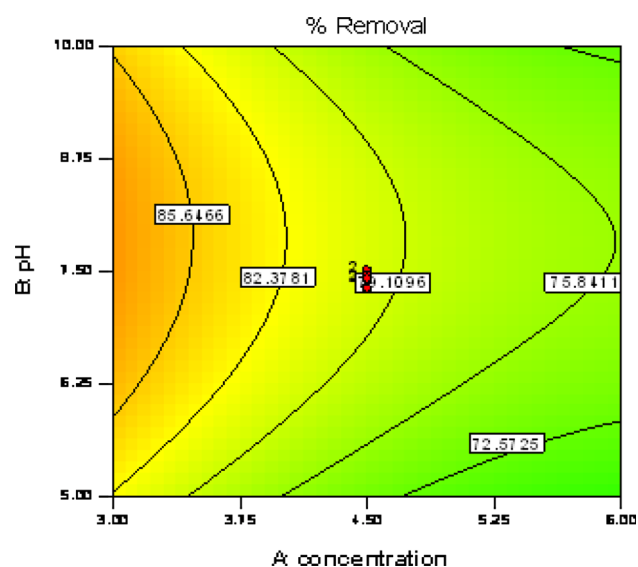
**Table 8** Analysis of variance (ANOVA) for percentage removal of Congo Red onto burned root of *E. crassipes* biomass

Source	Sum of squares	df	Mean square	F value	p value	Prob > F
Model	8793.39	14	628.10	43.11	<0.0001 significant	
A-conc.	1027.57	1	1027.57	70.53	<0.0001	
B-pH	49.08	1	49.08	3.37	0.0863	
C-dose	5920.79	1	5920.79	406.37	<0.0001	
D-time	103.30	1	103.30	7.09	0.0177	
AB	0.21	1	0.21	0.01	0.9057	
AC	354.95	1	354.95	24.36	0.0002	
AD	0.096	1	0.096	6.596E – 003	0.9363	
BC	0.83	1	0.83	0.057	0.8148	
BD	0.026	1	0.026	1.757E – 003	0.9671	
CD	8.1	1	8.18	0.56	0.4653	
A <sup>2</sup>	147.7	1	147.73	10.14	0.0062	
B <sup>2</sup>	718.8	1	718.89	49.34	<0.0001	
C <sup>2</sup>	345.62	1	345.62	23.72	0.0002	
D <sup>2</sup>	1.72	1	1.72	0.12	0.7357	
Residual	218.55	15	14.57			
Lack of Fit	214.63	14	15.33	3.91	0.3790	Not significant
Pure error	3.92	1	3.92			
Cor total	9011.94	29				

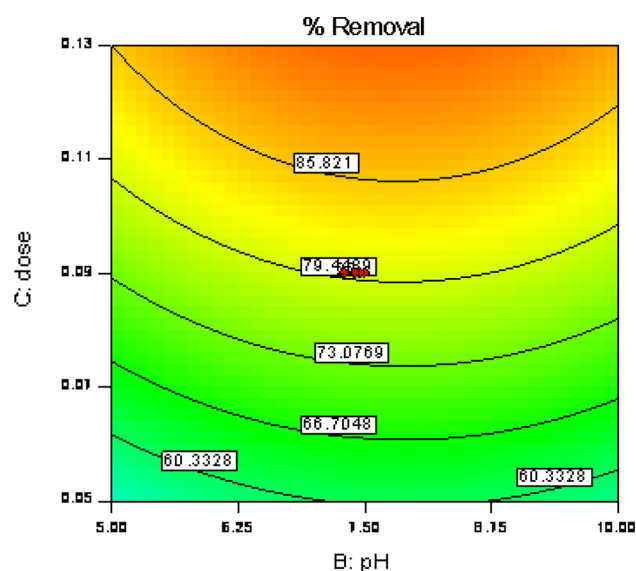
**Fig. 17** Comparison between the actual values and the predicted values of RSM model for adsorption of CRDesign-Expert® Software  
% RemovalColor points by value of  
% Removal:  
99  
32

and the  $R^2_{\text{Pred}}$  are measurements of the amount of variation around the mean and newly explained data, respectively. The selected quadratic model was validated by ANOVA, which provided evidence such as high  $F$  value (43.11), very low  $p$  value (<0.0001), non-significant lack of fit (3.91), as well as high values for coefficient of  $R$ -squared ( $R^2_d = 0.9757$ ), adjusted  $R$ -squared ( $R^2_{\text{Adj}} = 0.9531$ ),

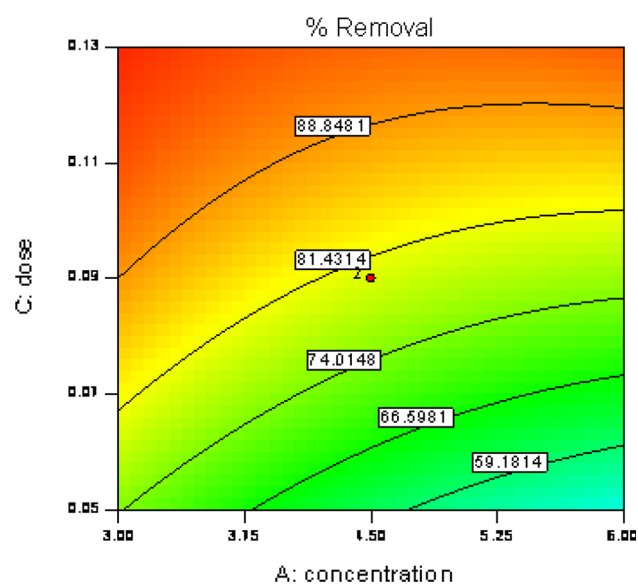
predicted  $R$ -squared ( $R^2_{\text{Pred}} = 0.8462$ ) and the adequate precision (25.138). Moreover, Fig. 17 shows the actual values versus predicted values of the CR adsorption onto *BREC*, which indicates an excellent agreement between the actual and predicted values. As observed, the validity (significance and adequacy) of the model was confirmed by the reasonable evidence (Figs. 18, 19, 20, 21).



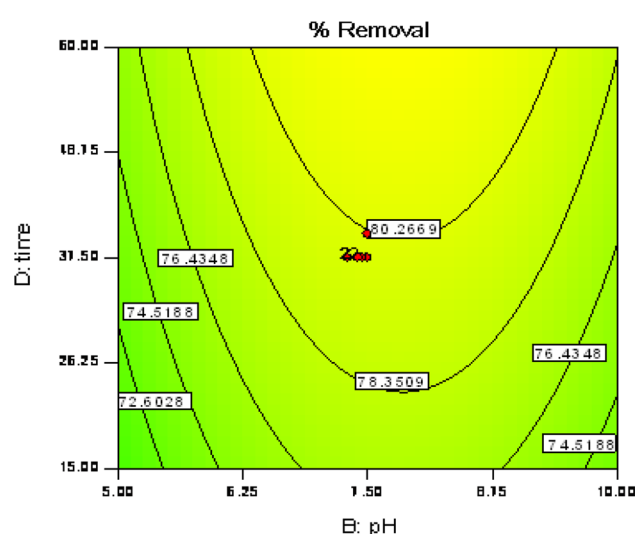
**Fig. 18** Contour plot showing the combined effect of initial concentration and pH on the removal of Congo Red by BREC



**Fig. 20** Contour plot showing the combined effect of biosorbent dose and pH on the removal of Congo Red by BREC



**Fig. 19** Contour plot showing the combined effect of initial concentration and dose on the removal of Congo Red by BREC



**Fig. 21** Contour plot showing the combined effect of contact time and pH on the removal of Congo Red by BREC

### Desorption studies

From the desorption study it was found that with increase in concentration of NaOH solutions, the desorption increased and more than 87 % of CR was desorbed from BREC using 5.0 M NaOH solution. The above washed adsorbents were reused to study further the change in removal efficiency of used adsorbents. Almost similar results were reported by Sadhukhan et al. (2014).

### Conclusions

The results of the present investigation showed that *E. crassipes* plant biomass could be a promising biosorbent for the removal of anionic Congo Red dye from aqueous medium. According to the ANOVA analysis, CR dye removal is highly affected by biomass dose, pH and most interacting effect was observed between pH with the other factors (biomass dose, initial concentration and contact time). The hierarchical quadratic model represents the adequacy of the response surface space based on the



adjusted determination coefficient ( $R_{adj}^2 = 0.9531$ ) and the adequate precision ratio (25.138). By using response surface methodology, the optimal conditions for CR removal were recorded as pH 7.0, 1.25 gm/L of biomass, 45 min contact time and 5 mg/L<sup>-1</sup> of CR concentration. The predicted removal efficiency achieved nearly 95 % of Congo Red using BREC biomass. Therefore, present research highlighted that the aquatic macrophyte could be efficient and effective biosorbent for other dyes also.

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